Recommended Specifications for Microchemical Apparatus

Micro-Kjeldahl Nitrogen

Committee for the Standardization of Microchemical Apparatus, Division of Analytical Chemistry, AMERICAN CHEMICAL SOCIETY

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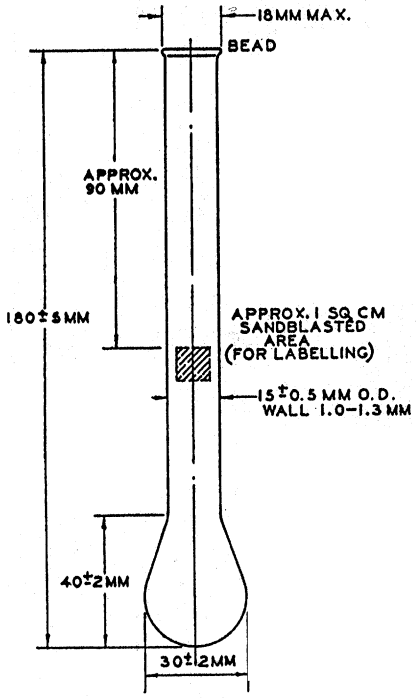


Figure 1. 10-Ml. Kjeldahl Flask

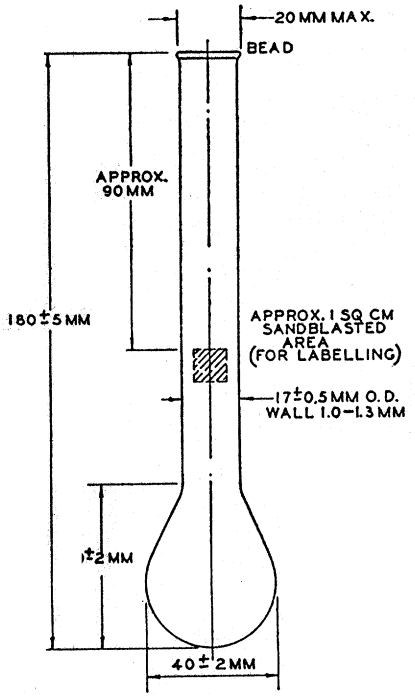


Figure 2. 30-Ml. Kjeldahl Flask

RECOMMENDED specifications for microchemical apparatus used in the carbon-hydrogen, Dumas nitrogen, halogen, and sulfur determinations (*2*) and the proposed program for the future (*1*) have been published. The present paper includes recommendations for the apparatus used in the Kjeldahl nitrogen determination and includes the following:

Kjeldahl digestion flasks, 10, 30, 30 (Soltys), and 100 ml.
Micro digestion rack
Manifold for micro digestion rack
Micro-Kjeldahl distillation apparatus, Pregl Type (Parnas-Wagner); electric steam generator
Micro-Kjeldahl distillation apparatus (one-piece model)

All recommendations have been made only after considerable experimental work by members of the committee.

Kjeldahl Digestion Flasks. Specifications for four types of flasks are recommended—10, 30, 30 (Soltys), and 100 ml. These are shown in Figures 1, 2, 3, and 4.

Micro Digestion Rack. The following specifications are recommended.

The micro-Kjeldahl digestion rack should be portable and consist of two parts, the flask heaters and the manifold support. The rack should provide for six Kjeldahl flasks, capacities 10 to 100 ml.

HEATERS. The source of heat may be either electricity or gas. The heaters must maintain a mixture of 2 ml. of sulfuric acid plus 1.30 grams of potassium sulfate at its boiling point (350° C.). The heaters must be sufficiently adjustable to provide for a mild digestion (low temperature) as well as distillation rates that will maintain refluxing of the acid into the neck of the flask. To avoid excessive heating of the flask necks, a shield is provided above the heaters. The shield should have circular openings not to exceed 26 mm. in diameter, directly above each heating element. The openings must be spaced 67 ± 7 mm. on centers. The rack must be provided with deflectors mounted under the burners to prevent overheating of the bench top.

Gas Heaters. The burners should be individually adjustable, with handles conveniently located at the front of the rack, and so constructed that they will be cool enough to manipulate even after long periods of operation. The burner must produce a nonluminous flame.

Electric Heaters. Electric heaters must perform satisfactorily at 100 volts. The controls must be conveniently located and should not become too hot to handle on continued op-

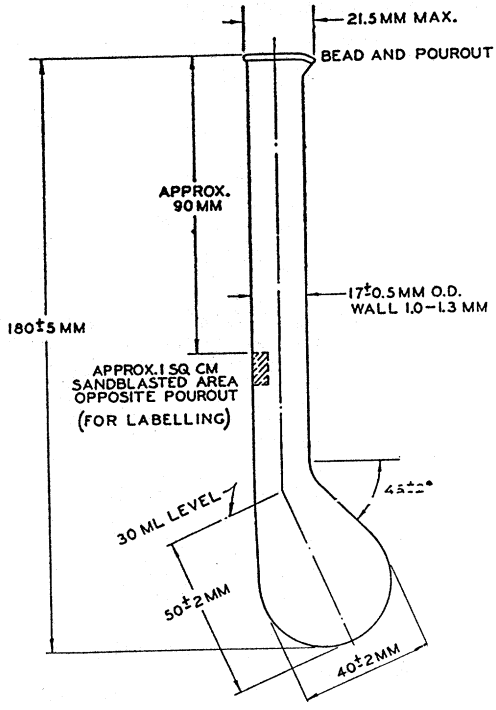


Figure 3. 30-Ml. (Soltys) Kjeldahl Flask

eration. Not more than two heaters should be operated from one set of controls.

MANIFOLD SUPPORT. This must hold the manifold securely, and also be adjustable, so that the manifold will support all the recommended sizes of flasks at the proper angle during digestion.

Manifold for Micro Digestion Rack. Figure 5 shows the recommended manifold. Any of the four types of Kjeldahl digestion flasks may be used with it.

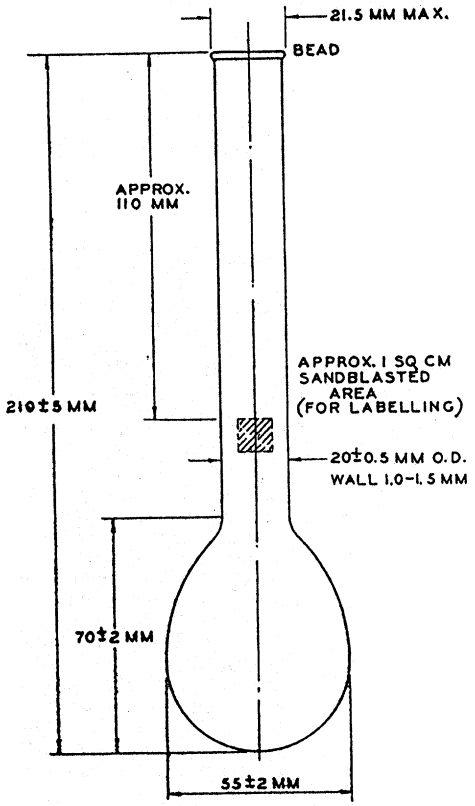


Figure 4. 100-Ml. Kjeldahl Flask

Micro-Kjeldahl Distillation Apparatus, Pregl Type (Parnas-Wagner). This apparatus, shown in Figures 6 and 7, is composed of the following parts:

Steam generator (2000-ml. capacity), *A*
Steam tube, *B*
Wiring assembly, *C*
Steam trap, *D*
Tube, *E*
Connecting tube, *F*
Distillation flask, *G*
Filling funnel, *H*
Condenser tube (glass or silver), *J*
Condenser jacket (West type), *K*
Drain, *L*

A new type of generator is recommended that provides closer control of steam generation than the usual gas-heated distillation flask. This all-glass steam generator, Figure 7*A*, is a wide-mouthed commercially available resin reaction kettle (Corning Glass Works, Corning, N. Y.). The interchangeable cover, with flat-ground rim and four ground-glass ⅌ stopper tubulations,

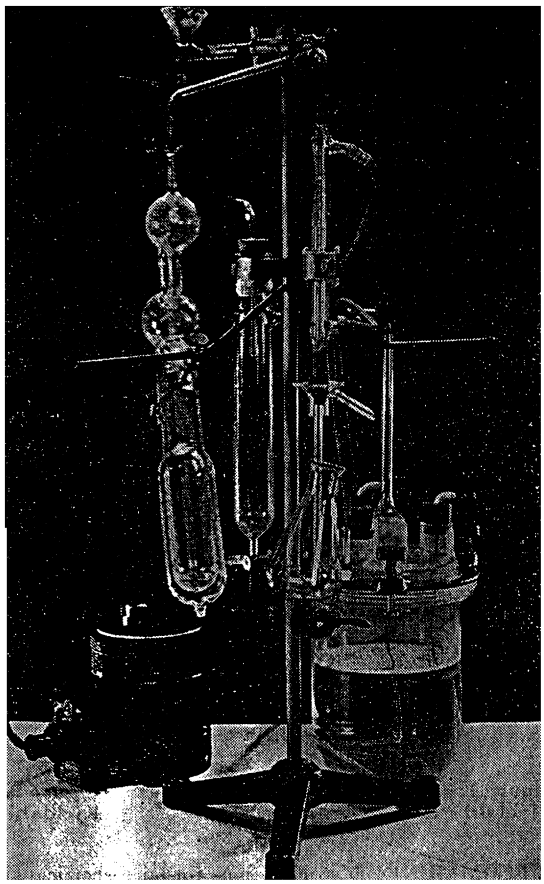


Figure 6. Micro-Kjeldahl Distillation Apparatus, Pregl Type (Parnas-Wagner)

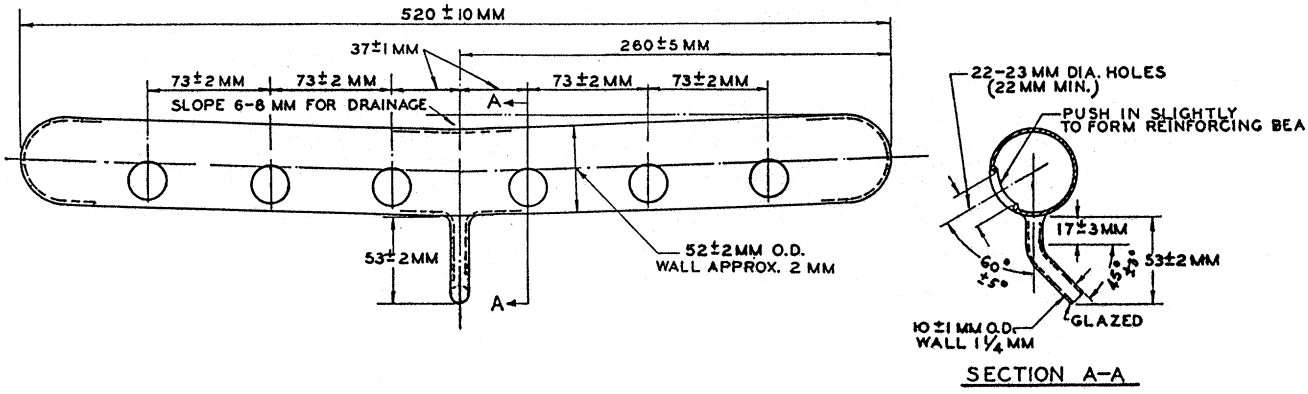
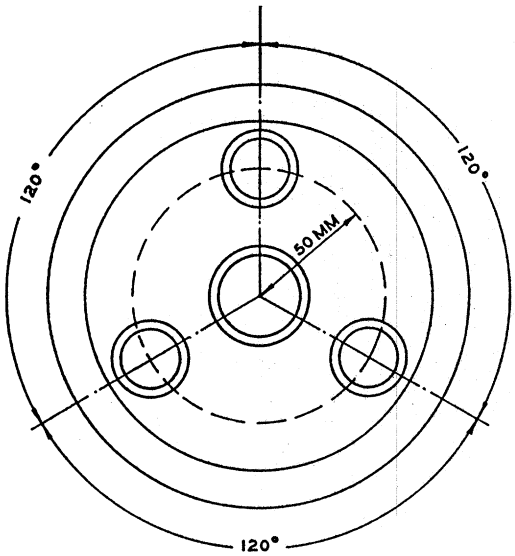


Figure 5. Manifold for Micro-Kjeldahl Digestion Rack



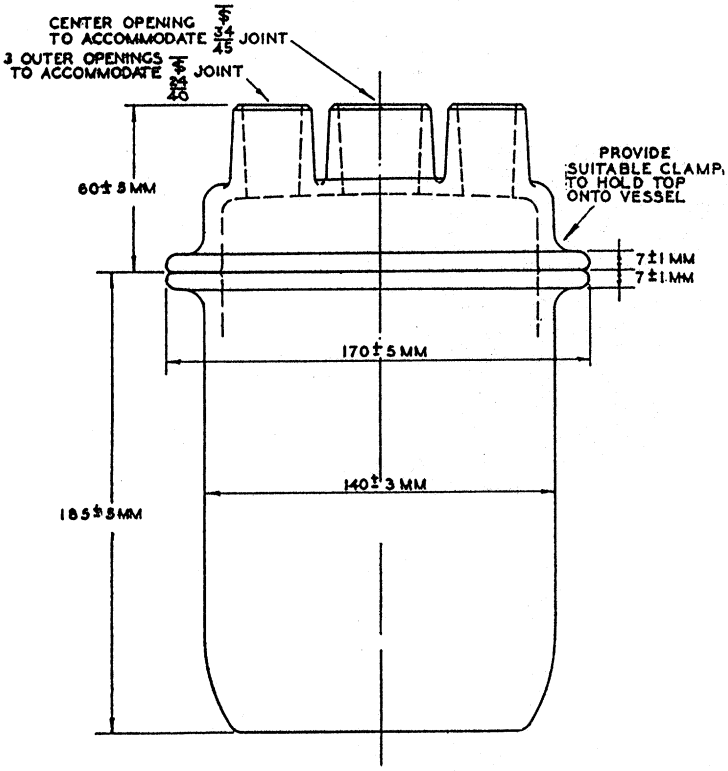


Figure 7A. Steam Generator, Micro-Kjeldahl Distillation Apparatus, Pregl Type (Parnas-Wagner)

is held in place with suitable clamps. To the center tubulation is attached the steam tube, Figure 7*B*. Two of the outer three tubulations are used for the leads of the electric heater assembly, Figure 7*C*. The remaining tubulation is used for a water inlet, or mounting a glass rod support for the resistance wire coil. The resistance of the heating spiral is about 16 ohms. The rate of steam generation is readily controlled by means of a 7.5-ampere variable autotransformer.

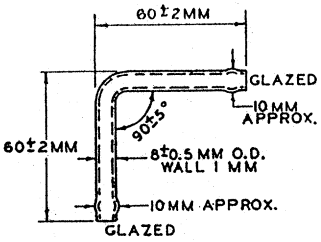


Figure 7F. Connecting Tube

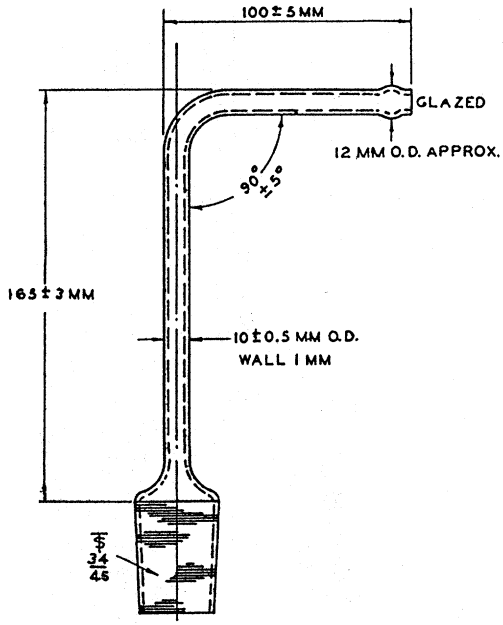


Figure 7B. Steam Tube

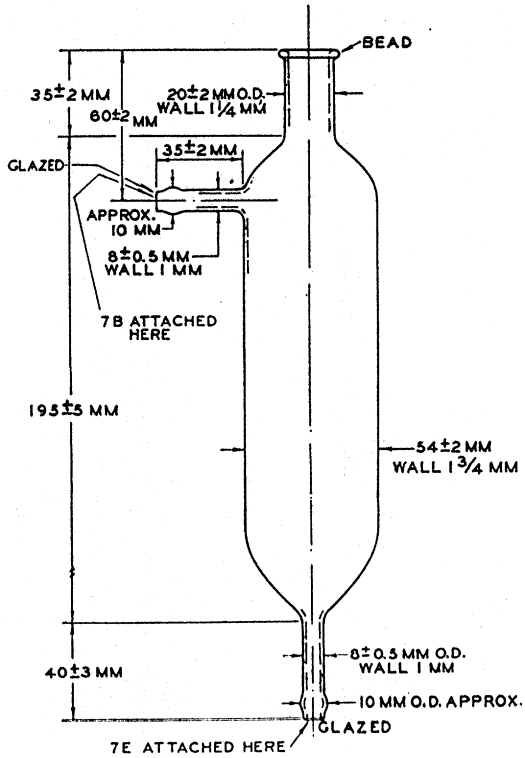


Figure 7D. Steam Trap

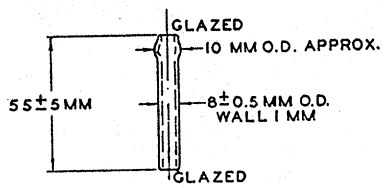


Figure 7E. Tube

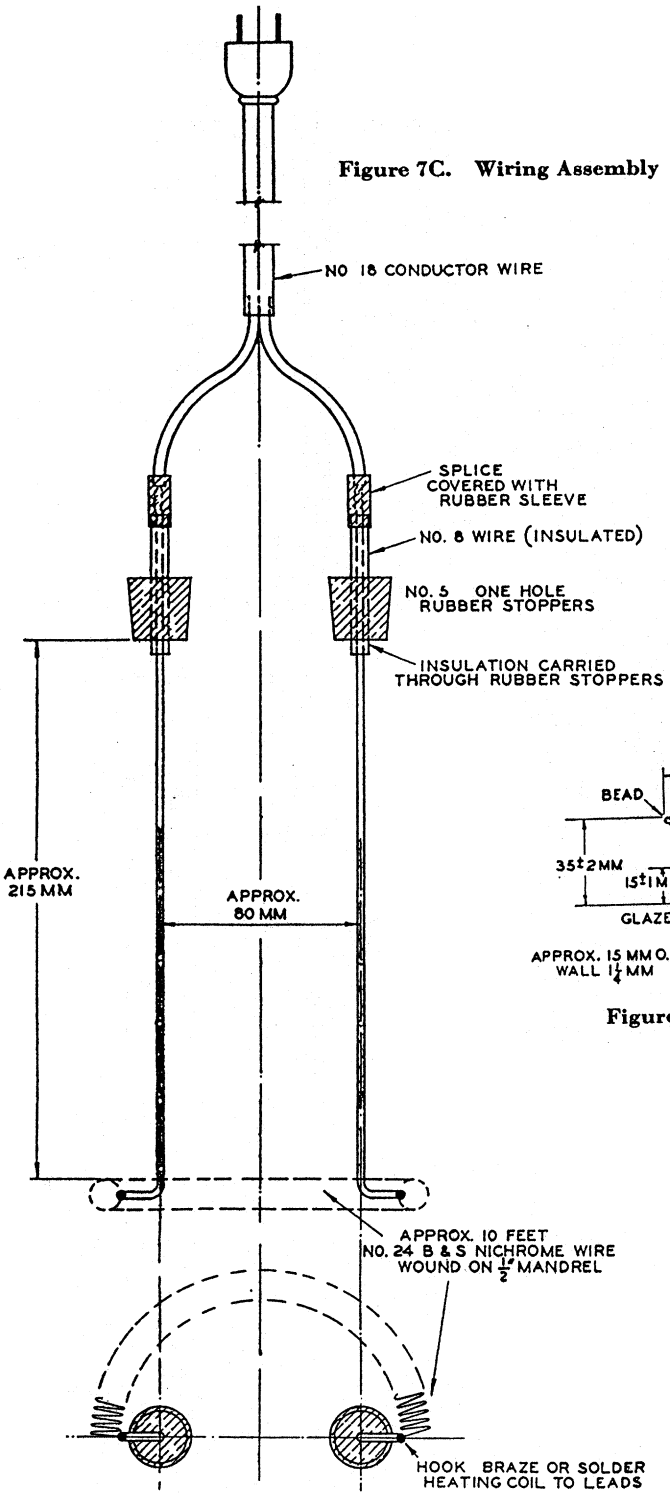


Figure 7C. Wiring Assembly

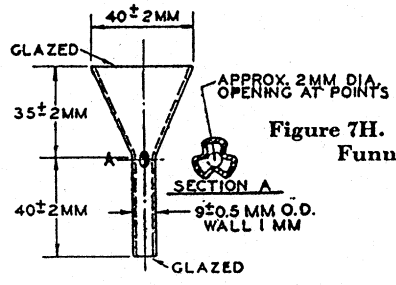


Figure 7H. Filling Funnel

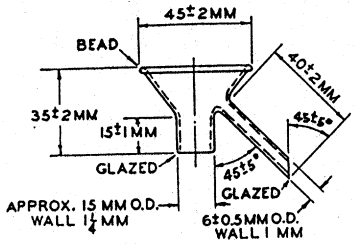


Figure 7L. Drain

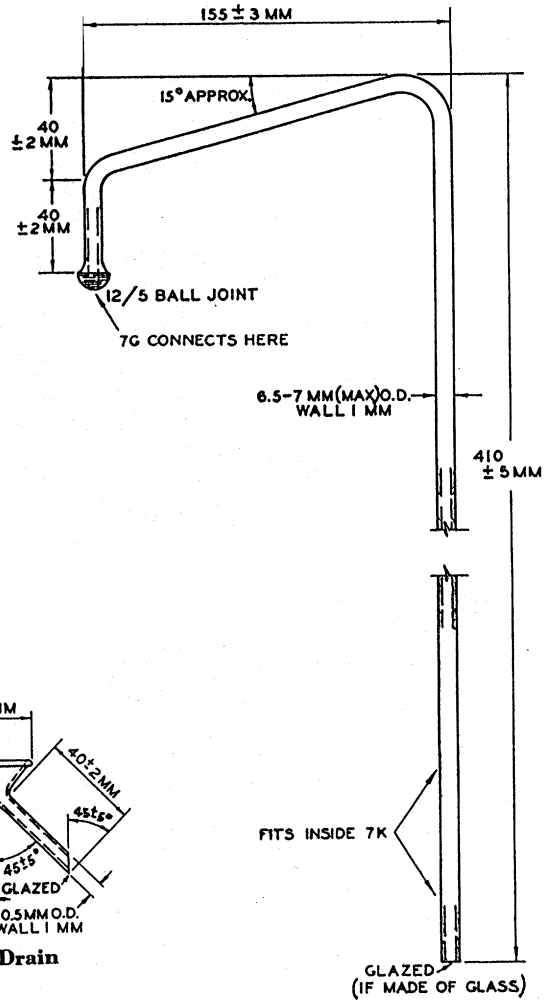


Figure 7J. Condenser Tube

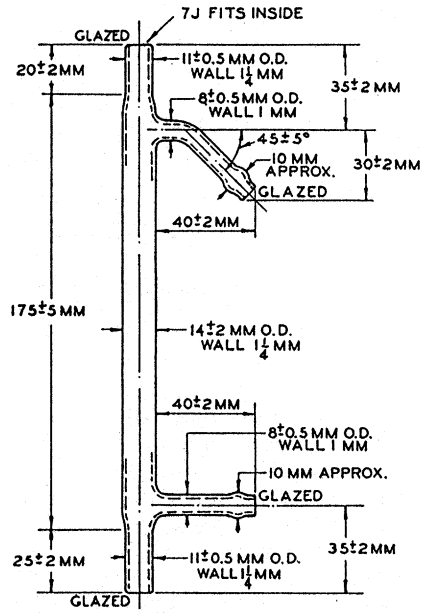


Figure 7K. Condenser Jacket (West Type)

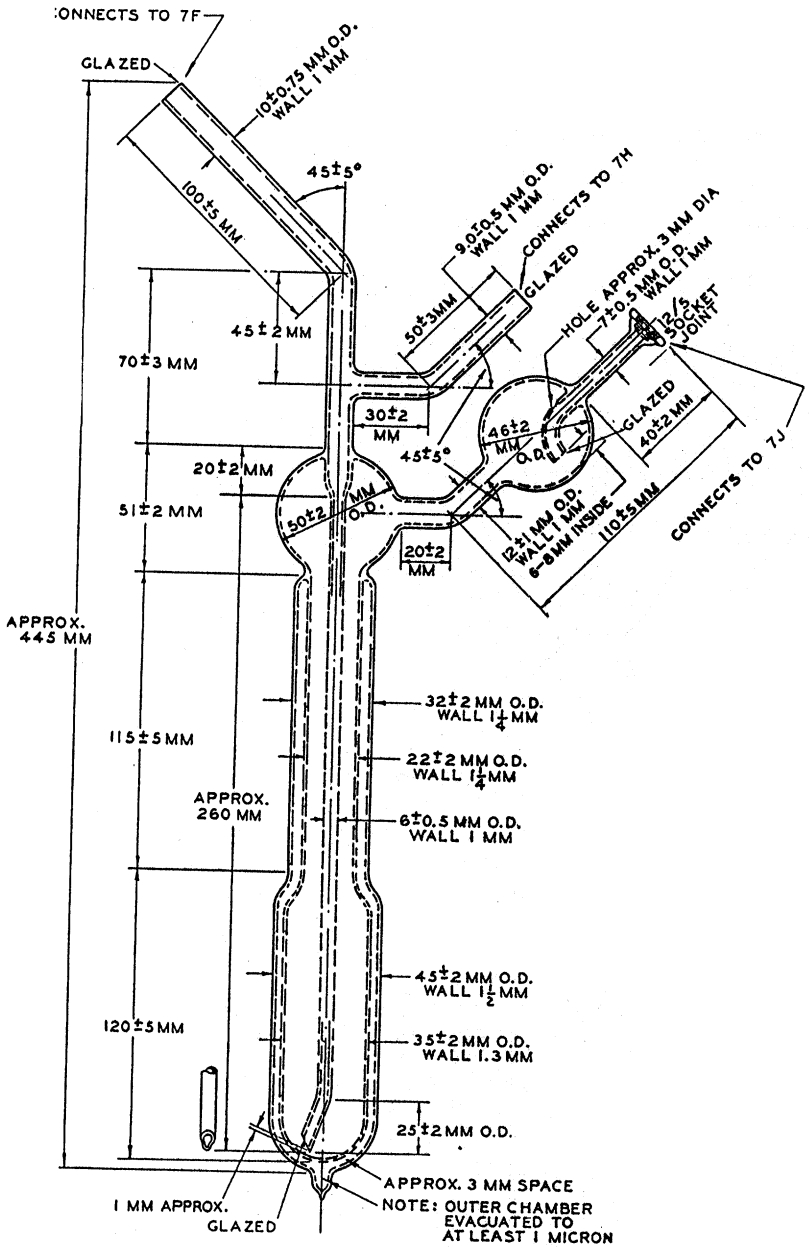


Figure 7G. Distillation Flask

This steam generator is also used with the Kjeldahl distillation apparatus, one-piece model (Figures 8 and 9), described below.

Changes have been made in the conventional designs of the distillation flask, Figure 7*G*, and the condenser tube, Figure 7 *J*. They include a ball and socket joint connection, which should be lubricated with stopcock grease and held together with a suitable clamp, and a West-type condenser.

Kjeldahl Distillation Apparatus. ONE-PIECE MODEL (Figures 8 and 9). An apparatus of this type, but having slightly different dimensions, has been commercially available for some time (Scientific Glass Apparatus Co., Inc., Bloomfield, N. J.). The origin of this apparatus is unknown, although attempts have been made to establish it. Because excellent results have been obtained with a one-piece model by many, including members of the committee, the design was studied. The specifications recommended here are the result of this work.

This revised model is used with the electric steam generator, Figure 7*A*, forming a sturdy, compact unit. The complete apparatus is shown in Figures 8 and 9.

The inner portion of this apparatus is heated by steam flowing through the system, in contrast to maintenance of the temperature by a vacuum jacket as in Figure 7*G*. The steam enters through the vertical tube extending almost halfway up the outer jacket at the left, surrounds the distillation flask proper (inner jacket), and passes into the small bent tube near the top at the right (above the sample funnel inlet), and then downward through the bent portion of the tube at the lower end of the inner jacket, and up into the center portion or the distillation flask. The two traps with T-shaped tubes hold back alkali spray.

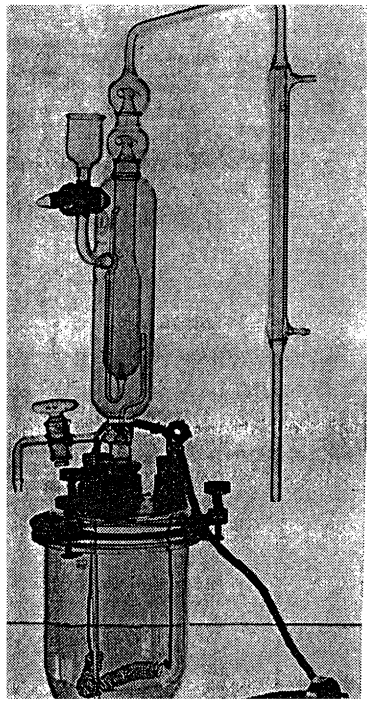


Figure 8. Micro-Kjeldahl Distillation Apparatus

One-piece model

OPERATION. With both stopcocks open, the sample is introduced through the funnel and the curved tube into the inner chamber or distillation flask. The alkali is added in like manner, displacing the acid portion upward. Steam is generated with the stopcocks on both the sample inlet funnel and the drainage tube closed, and the distillation is allowed to proceed.

Several methods of cleaning can be used. The following one has been found satisfactory, particularly when boiling chips are employed in the digestion:

While steam is being generated and with both stopcocks closed, the sample inlet funnel is filled with water and the tip of the condenser immersed in about 100 ml. of water contained in a beaker. Steam generation is stopped, and stopcock of the funnel is opened slowly, being closed before all the water has drained into the apparatus. Reduced pressure causes water to be sucked from the beaker into the apparatus, washing it. The stopcock in the drainage tube is opened to empty the liquid which has collected in the outer jacket, the steam generation is started again, and the procedure is repeated.

LITERATURE CITED

(1) Anal. Chem., 21, 651 (1949).

(2) Steyermark, Al, Alber, H. K., Aluise, V. A., Huffman, E. W. D., Kuck, J. A., Moran, J. J., and Willits, C. O., *Ibid.*, 21, 1555 (1949).

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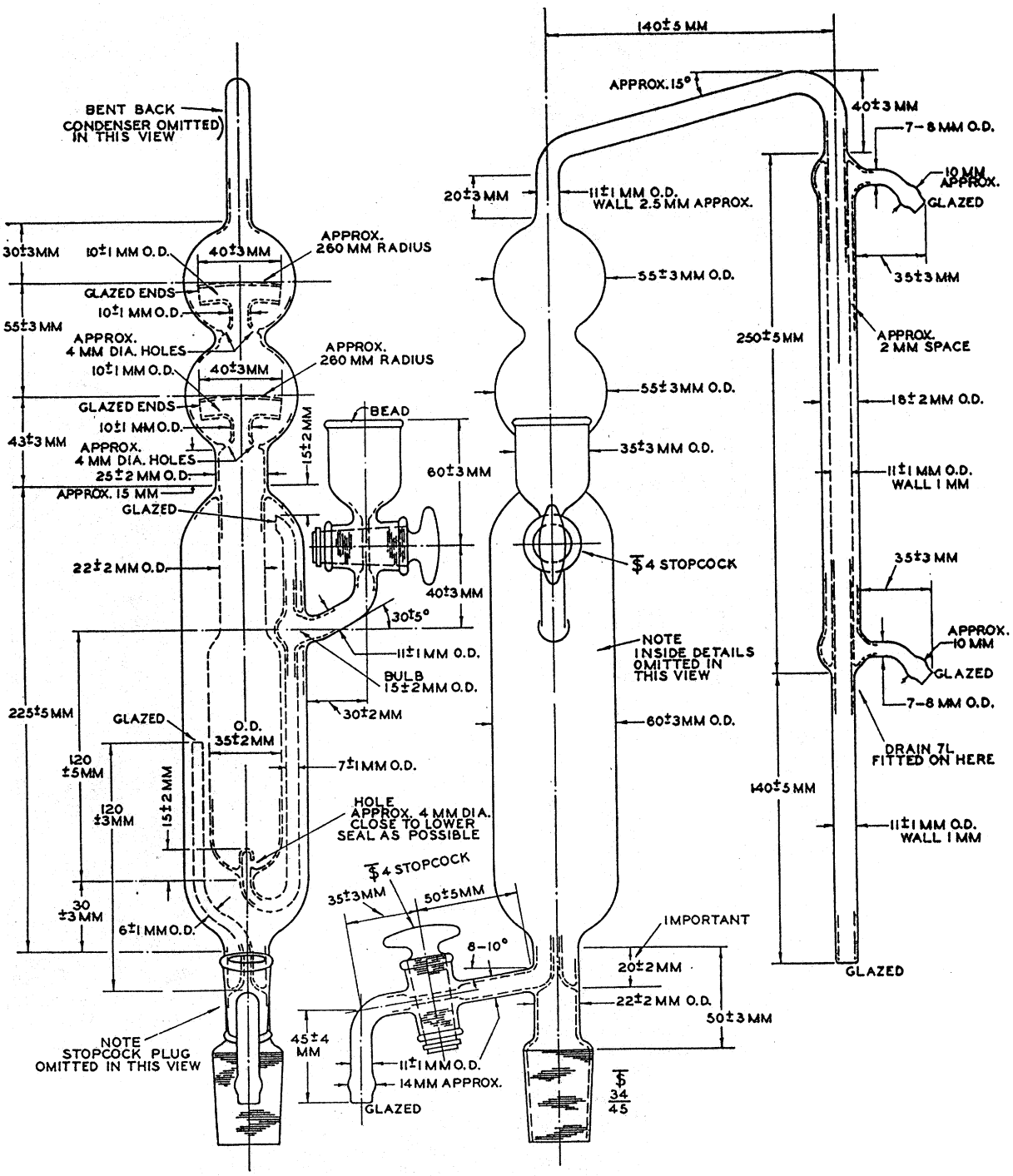

Figure 9. Kjeldahl Distillation Apparatus